DEVELOPMENT OF BATTERY SEPARATOR MATERIAL PROCESS

INTERIM REPORT

by

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I. SUMMARY

The preferred procedure for preparing sterilizable battery separator material involves grafting of low-density polyethylene film with acrylic acid, washing of the product with five percent potassium hydroxide, rinsing in water, drying, and crosslinking the dried material with divinylbenzene. The grafting and crosslinking procedures are initiated by irradiation from a cobalt-60 source.

Separator material produced by the above procedure and with the grafting carried out in a nitrogen atmosphere at 86 °F has an electrical resistance in the range of 6 to 15 milliohm-inch^2 with an average resistance of 10 milliohm-inch^2. Substantial amounts of material prepared by this procedure were shipped to the sponsor.

Separator material with a resistance value range of 2 to 6 milliohm-inch^2 and an average value of 4 milliohm-inch^2 has also been prepared using process modifications.

II. INTRODUCTION

A process for the preparation of a sterilizable battery separator material was developed by Radiation Applications, Inc. for the Jet Propulsion Laboratory. The process involved the crosslinking of low-density polyethylene film with divinylbenzene with subsequent grafting of the resultant film with acrylic acid. Both reactions were initiated by radiation from a cobalt-60 source. Selected samples of battery separator material performed well in silver-zinc batteries, but it was considered necessary to improve the reproducibility and uniformity of the separator material

The purpose of this experimental program was to study the basic parameters of the process and optimize the procedure to yield a battery separator material with a uniformly low electrical resistance in 40% potassium hydroxide solution. Initially, the work was to be directed toward the acrylic acid grafting of the divinylbenzene-crosslinked polyethylene film The parameters to be studied were irradiation dose rate, total irradiation dose, temperature during irradiation, film washing procedures, and the effect of the presence of oxygen during irradiation.

The original test plan for studying the grafting procedure is given in the Mid-Program Report dated February 15, 1967. This plan consisted of a few preliminary experiments and a one-half replicate of a 2^5 factorial

experiment for studying the parameters which were felt to be pertinent to the procedure for grafting the divinylbenzene-crosslinked polyethylene film.

The results of the preliminary experiments indicated that a superior product was produced if the polyethylene film was grafted prior to crosslinking. As a consequence, the factorial experiment was revised to accommodate this procedure. The design of the modified factorial experiment is shown in Table 1.

The desired properties of the battery separator material are a maximum electrical resistance of 120 milliohm-inch^2 in 40% potassium hydroxide solution and a minimum wet tensile strength of 700 psi.

III. EXPERIMENTAL

A. Nomenclature

Throughout this program, 30-foot rolls of low-density polyethylene film were modified. To identify the various parts of the rolls which were analyzed, the following procedures and nomenclature were used.

One-foot samples of film were taken from various parts of the rolls of modified polyethylene film for analysis. These samples were identified by roll number, position along the length of the roll, and position across the width of the roll. The designation "15-1, middle" represents roll number 15, the first foot of the roll (outermost part of roll), and the center part across the sample, respectively. The nomenclature applies to the modified film, and the position along the roll may be greater than 30 as the modification causes an increase in the length of the film

When both crosslinking and grafting procedures are performed on the same roll of film, the innermost part of the roll from the first treatment becomes the outermost part for the second treatment during the rerolling operation. All sample nomenclature is based on the position of the film during grafting.

B Radiation Configuration and Dosimetry

The radiation configuration and the dosimetry procedures were the same as described in the Mid-Program Report dated February 15, 1967.

C. Preliminary Experiments

The general procedures for grafting and crosslinking of the low-density polyethylene film are given in the Appendix of this report.

The preliminary experiments reported in the Mid-Program Report indicated that grafting of the polyethylene film with subsequent crosslinking produced a product far superior to that obtained when the polyethylene film was crosslinked first. The electrical resistance values of the product were considerably lower and much more uniform when grafting was carried out prior to crosslinking.

To determine the reproducibility of the improved procedure and to prepare samples for the Sponsor, ten additional 30-foot rolls of low-density polyethylene were grafted and then crosslinked. The results are tabulated in Table 2. As reported in the Mid-Program Report, grafting was erratic in the outermost four feet of each roll, but resistance values of the remainder of each roll were well below the maximum permissible value and fairly uniform.

All of the aforementioned grafting with acrylic acid was carried out in air at a dose rate of 0.021 Mrad per hour for a total dose of 1.430 Mrad. To determine if a lower total dose would produce sufficient grafting, one roll of polyethylene film was grafted with acrylic acid in the presence of air at a dose rate of 0.025 Mrad for a total dose of 0.550 Mrad. The outermost seven to eight feet of the roll showed

erratic grafting, while the remainder of the roll showed uniformly low electrical resistance values. These data are shown in Table 3.

D. Factorial Experiment

The data obtained for the runs listed in the factorial experiment are shown in Tables 4 through 19. A summary of the electrical resistance values is given in Table 20. In all cases, the compositions of the grafting solution and crosslinking solution, when required, were held constant. Gas pockets were removed from the rolls by alternately lowering and raising the pressure in the reaction vessel. The grafted film was soaked for one hour at 80°C in five percent potassium hydroxide solution followed by a soak for one hour in distilled water at 80°C. The wet films were permitted to dry in air at ambient conditions (about 75°F and 50% relative humidity). Crosslinked films were rinsed with benzene after crosslinking. Samples for resistance and tensile strength measurements were permitted to soak in 40% potassium hydroxide solution for at least 18 hours before the measurements were made.

Analysis of the data from the factorial experiment indicates the following:

1. The higher temperature favors lower and more uniform electrical resistance values throughout the roll.

2. A nitrogen atmosphere favors lower and more uniform electrical resistance values throughout the roll.

3. The lower dose rate produces film which is grafted throughout the roll in both air and nitrogen atmospheres. With the high dose rate, grafting usually does not occur in air at ambient temperature on the outermost few feet. If the outermost few feet of the roll are not considered, dose rate in the range studied has little effect on electrical resistance.

4. Total dose, within the range studied, has little effect on electrical resistance.

5. The degree of crosslinking obtained with the procedure employed has little, if any, effect on electrical resistance.

6. Within experimental error, the total dose, dose rate, and crosslinking have little effect on tensile strength. The higher temperature as well as a nitrogen atmosphere decreases the tensile strength. This is probably due to an increased degree of grafting. However, the tensile strengths obtained in all cases are acceptable.

7. A combination of the higher temperature and a nitrogen atmosphere produced modified film which was difficult to recover from the roll. It appeared to be overgrafted, and it is believed that a much lower total dose under these conditions would produce a good battery separator material of low electrical resistance.

In no case was an exotherm produced during grafting in the presence of air at 77^{o}F (Table 21). However, grafting in the presence

of air at 125°F usually exhibited an exotherm, although the exotherm was not as great as obtained with a nitrogen atmosphere at this temperature.

E. Effect of Sterilization

The effect of sterilizing a number of samples of modified film obtained during the statistical experiment is shown in Table 22. The samples were picked in a random fashion as only one sample was chosen for each set of conditions used in the statistical experiment. Most of the results were typical. However, the resistance of one sample (52-13) which had a relatively high resistance of 109 milliohm $inch^2$ decreased to 9 milliohm $-inch^2$. This sample also had a relatively large increase in thickness on sterilization.

F. Miscellaneous Experiments

To check the reproducibility of the grafting of battery separator material at ambient temperature in a nitrogen atmosphere and supply material for use by the Sponsor, fifteen additional rolls of polyethylene film were grafted and crosslinked The reproducibility of the electrical resistance (Tables 23 and 24) was excellent, and the material was shipped to the Sponsor. The higher ambient grafting temperature (86° and 89°F) caused a decrease in the average resistance (11 milliohm $-inch^2$) from that obtained at 77°F (22 milliohm $-inch^2$, Table 5).

Excellent reproducibility of electrical resistance was also obtained when the grafting was carried out at 125°F in the presence of air (Tables 13, 25 and 26).

As higher temperatures and nitrogen atmosphere improved the grafting of low-density polyethylene film, attempts were made to graft divinylbenzene-crosslinked polyethylene film with acrylic acid at 90°F in nitrogen (Table 27), at 125°F in air (Table 28), and at 125°F in nitrogen (Table 29). In nitrogen at 90°F and in air at 125°F, grafting occurred only on the innermost part of the roll, while about one-half of the roll grafted in nitrogen at 125°F.

Washing of the crosslinked film in boiling five percent potassium hydroxide solution permitted grafting (90°F in nitrogen) to occur throughout the roll (Table 30); however, the resistance values were high throughout most of the roll. Lower resistance values would probably be obtained in nitrogen at 125°F. It appears that material from the divinylbenzene solution which acts as an inhibitor remains in the film after crosslinking, and satisfactory battery separator material could probably be prepared from the crosslinked film if a proper washing procedure is used. However, grafting with subsequent crosslinking is the preferred procedure.

In determining the effect of acrylic acid concentration in the grafting solution, the volume of grafting solution and the ratio of

acrylic acid to carbon tetrachloride were held constant. Very little, if any, grafting occurred when the acrylic acid concentration was five weight percent (Table 31). Ten weight percent acrylic acid (Table 32) was borderline, while twenty weight percent (Table 34) was somewhat better than fifteen weight percent (Table 33) and was approximately equivalent to twenty-five weight percent (Tables 23 and 24).

The initial study of the effect of chain terminator (carbon tetrachloride) concentration was inconclusive because of erratic results. The high temperature used, the high erratic exotherm, and the low boiling point of the benzene solvent were responsible for the erratic results. In many cases, most of the benzene boiled off. When this occurred, the film and paper could be separated, if at all, only with extreme difficulty. Replacement of part of the benzene with xylene was of some help, while replacement of all of the benzene with xylene permitted the film and paper to be separated readily The data obtained for the aforementioned runs is tabulated in Tables 35 through 43. Future work on the effect of chain terminator will be done at lower temperatures and in the presence of nitrogen.

IV. CONCLUSIONS

Grafting of low-density polyethylene film with acrylic acid followed by crosslinking with divinylbenzene yields a battery separator material superior to that produced by reversing the order of the grafting and crosslinking. The product is much more uniform, and the electrical resistance in forty-percent potassium hydroxide solution is well below the desired maximum value.

From the experimental work conducted to date, the preferred preparative procedure for the sterilizable battery separator material involves grafting of low-density polyethylene film with acrylic acid in a nitrogen atmosphere at 80°-90°F and crosslinking the potassium salt of the product with divinylbenzene.

More experimental work should be conducted to complete the optimization of the preparative procedure.

V. FUTURE WORK

Optimization of the grafting procedure will be completed. This will include optimization of acrylic acid and chain terminator concentrations.

The effect of acrylic acid solvents on grafting will be determined.

The potassium salt of the grafted film will be crosslinked by irradiation in an electron beam, and the product will be compared with the material obtained by divinylbenzene crosslinking.

Ten thousand feet of battery separator material will be prepared and supplied in 500-foot lengths. A device for neutralizing and washing large quantities of film will be designed and constructed for use in preparing this large quantity of film.

To date, the degree of grafting, the degree of crosslinking, and the potassium content of the battery separator material have not been determined. These will be determined on many of the films which are believed to have different compositions, and, if possible, they will be correlated with performance of the separator material in batteries.

Other acids and chain terminators will be evaluated for grafting polyethylene film.

Other crosslinking materials will be evaluated.

TABLE 1. REVISED FACTORIAL EXPERIMENT

		Standard Dose Rate				1/2 Standard Dose Rate			
		Std. Dose		1/2 Std. Dose		Std. Dose		1/2 Std. Dose	
		O_2	N_2	O_2	N_2	O_2	N_2	O_2	N_2
Grafted Only	Temp 1	X			X		X	X	
	Temp 2		X	X		X			X
Grafted then Crosslinked	Temp 1		X	X		X			X
	Temp 2	X			X		X	X	

Standard Dose Rate = 0.021 Mrad/hr

Standard Dose = 1.43 Mrad

Temp 1 = Ambient (about 75°F)

Temp 2 = 125°F

Standard washing procedure = 1 hr. in KOH at 80°C
1 hr. in H_2O at 80°C

Properties to be determined: (1) Electrical resistance at 25°C, ± 1°C
(2) Tensile strength (wet)
(3) Elongation

Crosslinking: Dose Rate: 0.025 Mrad
Total Dose: 0.550 Mrad
Temperature: Ambient (about 75°F)

TABLE 2. PROPERTIES OF POLYETHYLENE FILM WHICH WAS GRAFTED* AND THEN CROSSLINKED** IN PRESENCE OF AIR

Sample	Tensile Strength, psi	Elongation, %	Thickness, mil	Resistance, milliohm-inch^2
20-6, Top	1187	>100	1.5	16
	1575	>100	1.5	15
20-12, Middle	1320	>100	1.5	17
	1480	>100	1.5	22
20-21, Bottom	1465	>100	1.2	26
	1465	>100	1.2	23
20-32, Top	1360	>100	1.5	19
	1540	>100	1.5	-
23-6, Top	1175	63	1.5	22
	1500	90	1.5	22
23-15, Middle	1305	94	1.6	31
	1390	>100	1.5	24
23-24, Bottom	1375	>100	1.6	20
	1465	>100	1.5	20
23-34, Top	1284	>100	1.5	27
	1560	>100	1.2	19
28-5, Top	-	-	-	20
28-1, Bottom	1505	>100	1.0	283
28-35, Bottom	1270	>100	1.3	46
	-	-	-	49

TABLE 2. (Continued)

Sample	Tensile Strength, psi	Elongation %	Thickness, mil	Resistance, milliohm-inch^2
29-6, Bottom	1495	>100	1.4	15
29-24, Middle	1650	>100	1.4	46
30-11, Bottom	1430	>100	1.5	15
30-36, Top	1415	>100	1.4	17
31-7, Top	1570	80	1.4	16
31-30, Bottom	1540	80	1.5	72
32-6, Bottom	1260	>100	1.5	20
32-33, Middle	1440	90	1.3	23
33-7, Middle	1390	>100	1.5	18
33-35, Bottom	1450	98	1.4	21
34-6, Middle	1260	90	1.4	12
34-33, Middle	1480	>100	1.3	16
35-6, Bottom	845	80	1.3	27
35-35, Middle	1455	>100	1.4	17

* Dose rate of 0.021 Mrad per hour for a total dose of 1.430 Mrad
** Dose rate of 0.025 Mrad per hour for a total dose of 0.550 Mrad.

TABLE 3. PROPERTIES OF POLYETHYLENE FILM WHICH WAS GRAFTED* IN PRESENCE OF AIR

Sample	Tensile Strength, psi	Elongation %	Thickness, mil	Resistance, milliohm-inch^2
19-1, Top	2090	>100	1.0	>3000
	2420	>100	1.0	>3000
19-12, Middle	1470	>100	1.3	17
	730	>100	1.5	11
19-23, Bottom	1175	>100	1.7	11
	1510	>100	1.6	11
19-34, Top	730	>100	1.5	14
	1610	>100	1.3	12

* Grafted for 22 hours at a rate of 0.025 Mrad per hour.

TABLE 4. SAMPLE NUMBERS 36 AND 37

GRAFTED ONLY

Experimental Conditions for Grafting:
Dose Rate: 0.021 Mrad/hr
Total Dose: 1.430 Mrad
Temperature: 77°F
Atmosphere: Air

RESULTS:

Sample	Tensile Strength, psi	Elongation, %	Thickness, mil	Resistance, milliohm-inch^2	Resistance, milliohm-inch^2/mil
36-1, Top	900	>100	1.1	>3000	-
	1330	>100	1.2	>3000	-
36-32, Top	1690	>100	1.3	17	13
	1440	>100	1.3	17	13
37-1, Bottom	1440	>100	1.3	16	12
	1605	>100	1.3	12	9
37-11, Top	1970	>100	1.0	28	28
	2030	>100	1.0	40	40
37-20, Middle	2070	>100	1.2	48	40
	1695	>100	1.2	58	48
37-32, Top	1130	>100	1.3	16	12
	1160	>100	1.4	16	11
*Average	1623	-	1.2	27	23
*Range	1130-2070	-	1.0-1.4	12-58	11-48

* Does not include high resistance specimens.

TABLE 5. SAMPLE NUMBERS 38 AND 39

GRAFTED AND CROSSLINKED

Experimental Conditions for Grafting:
Dose Rate: 0.021 Mrad/hr
Total Dose: 1.430 Mrad
Temperature: 77°F
Atmosphere: Nitrogen

Experimental Conditions for Crosslinking:
Dose Rate: 0.025 Mrad/hr
Total Dose: 0.550 Mrad
Temperature: 77°F
Atmosphere: Air

RESULTS:

Sample	Tensile Strength, psi	Elongation, %	Thickness, mil	Resistance, milliohm-inch^2	Resistance, milliohm-inch^2/mil
38-1, Top	1175	87	1.5	30	20
	1140	75	1.4	9	6
38-32, Bottom	1320	99	1.5	27	18
	1305	97	1.6	19	12
39-1, Top	1350	98	1.3	11	8
	1255	80	1.4	11	8
39-9, Middle	1605	>100	1.3	38	29
	1510	99	1.2	29	24
39-17, Bottom	1375	92	1.6	27	17
	1250	98	1.8	25	14
39-25, Top	1255	>100	1.4	19	14
	1140	90	1.4	20	14
39-33, Middle	1395	93	1.5	20	13
	1395	98	1.5	27	18
Average	1319	-	1.5	22	15
Range	1140 1605	-	1.2 - 1.8	9-38	6-29

TABLE 6. SAMPLE NUMBERS 44 AND 45

GRAFTED ONLY

Experimental Conditions for Grafting:
Dose Rate: 0.021 Mrad/hr
Total Dose: 0.671 Mrad
Temperature: 77°F
Atmosphere: Nitrogen

RESULTS:

Sample	Tensile Strength, psi	Elongation, %	Thickness, mil	Resistance, milliohm-inch^2	Resistance, milliohm-inch^2/mil
44-1, Middle	755	64	1.6	12	8
	870	82	1.5	12	8
44-10, Top	1250	>100	1.6	12	7
	1395	>100	1.5	21	14
44-19, Bottom	840	60	1.5	16	11
	935	72	1.6	11	7
44-27, Middle	970	70	1.7	19	11
	865	40	1.6	18	11
44-33, Bottom	1170	85	1.6	14	9
	1030	73	1.5	14	9
45-1, Middle	1260	>100	1.7	10	6
	1255	>100	1.5	9	6
45-34, Top	1170	80	1.5	13	9
	1535	>100	1.5	7	5
Average	1093	-	1.6	13	9
Range	755-1535	-	1.5 - 1.7	9-21	5-14

TABLE 7. SAMPLE NUMBERS 46 AND 47

GRAFTED AND CROSSLINKED

Experimental Conditions for Grafting:
Dose Rate: 0.021 Mrad/hr
Total Dose: 0.671 Mrad
Temperature: 77°F
Atmosphere: Air

Experimental Conditions for Crosslinking:
Dose Rate: 0.025 Mrad/hr
Total Dose: 0.550 Mrad
Temperature: 77°F
Atmosphere: Air

RESULTS:

Sample	Tensile Strength, psi	Elongation, %	Thickness, mil	Resistance, milliohm-inch^2	Resistance, milliohm-inch^2/mil
46-1, Top	1265	>100	1.0	>3000	>3000
	1265	>100	1.0	>3000	>3000
46-34, Bottom	1160	80	1.7	15	9
	1305	>100	1.6	18	11
47-1, Top	2160	>100	1.1	>3000	>3000
	2160	>100	1.1	>3000	>3000
47-9, Middle	1430	84	1.5	36	24
	1700	>100	1.5	37	25
47-17, Bottom	1035	75	1.7	13	8
	1340	90	1.6	19	12
47-25, Top	1165	75	1.7	21	12
	1340	90	1.6	19	12
47-34, Middle	1280	88	1.5	16	11
	1250	80	1.5	18	12
*Average	1277	-	1.6	20	13
*Range	1035 1700	-	1.5-1.7	10-37	11-25

* Does not include high resistance specimens.

TABLE 8. SAMPLE NUMBERS 48 AND 49

GRAFTED ONLY

Experimental Conditions for Grafting:
Dose Rate: 0.0125 Mrad/hr
Total Dose: 1.700 Mrad
Temperature: 77°F
Atmosphere: Nitrogen

RESULTS:

Sample	Tensile Strength, psi	Elongation, %	Thickness, mil	Resistance, milliohm-inch^2	Resistance, milliohm-inch^2/mil
48-1, Middle	920	>100	1.5	16	11
	735	83	1.5	20	13
48-34, Bottom	1160	98	1.8	20	11
	1165	97	1.7	18	11
49-1, Bottom	1465	>100	1.5	17	11
	1535	>100	1.5	17	11
49-10, Top	1260	96	1.7	28	16
	1530	>100	1.4	26	19
49-19, Bottom	1575	>100	1.5	25	17
	1100	95	1.8	34	19
49 26, Middle	1390	>100	1.5	26	17
	1390	>100	1.5	29	19
49 34, Top	1360	97	1.5	30	20
	1210	75	1.5	24	16
Average	1271	-	1.6	24	15
Range	735-1575	-	1.4-1.8	16-34	11-20

TABLE 9. SAMPLE NUMBERS 50 AND 51

GRAFTED AND CROSSLINKED

Experimental Conditions for Grafting:
Dose Rate: 0.0125 Mrad/hr
Total Dose: 1.700 Mrad
Temperature: 77°F
Atmosphere: Air

Experimental Conditions for Crosslinking:
Dose Rate: 0.025 Mrad/hr
Total Dose: 0.550 Mrad
Temperature: 77°F
Atmosphere: Air

RESULTS:

Sample	Tensile Strength, psi	Elongation, %	Thickness, mil	Resistance, milliohm-inch^2	Resistance, milliohm-inch^2/mil
50-1, Middle	1215	70	1.4	20	14
	1375	75	1.5	21	14
50-34, Top	1465	87	1.5	36	24
	1430	92	1.5	71	47
51-1, Top	1430	>100	1.5	21	14
	1595	>100	1.4	20	14
51-9, Middle	1855	>100	1.4	66	47
	1850	>100	1.5	65	43
51-17, Bottom	1450	>100	1.6	29	18
	1580	>100	1.5	35	23
51-25, Top	1535	>100	1.7	62	36
	1465	>100	1.5	64	43
51-34, Middle	1650	>100	1.4	35	25
	1695	>100	1.4	55	39
Average	1542	-	1.5	43	25
Range	1215-1695	-	1.4-1.7	20-71	14-47

TABLE 10. SAMPLE NUMBERS 52 AND 53

GRAFTED ONLY

Experimental Conditions for Grafting:
Dose Rate: 0.0125 Mrad/hr
Total Dose: 0.800 Mrad
Temperature: 77°F
Atmosphere: Air

RESULTS:

Sample	Tensile Strength, psi	Elongation, %	Thickness, mil	Resistance, milliohm-inch^2	Resistance, milliohm-inch^2/mil
52-1, Bottom	1255	80	1.5	11	7
	855	76	1.6	13	8
52-13, Middle	2220	>100	1.2	133	111
	2160	>100	1.2	109	91
	-	-	-	71	-
	-	-	-	36	-
52-23, Top	2500	>100	1.3	15	12
	1440	>100	1.3	28	22
52-34, Middle	1845	>100	1.4	29	21
	1570	>100	1.4	23	16
53-1, Middle	920	>100	1.5	13	9
	845	82	1.5	10	7
53-34, Top	1480	>100	1.6	12	8
	1685	>100	1.5	12	8
Average	1565	-	1.4	34	27
Range	885-2500	-	1.2-1.6	10-133	8-111

TABLE 11. SAMPLE NUMBERS 54 AND 55

GRAFTED AND CROSSLINKED

Experimental Conditions for Grafting:
Dose Rate: 0.0125 Mrad/hr
Total Dose: 0.800 Mrad
Temperature: 77°F
Atmosphere: Nitrogen

Experimental Conditions for Crosslinking:
Dose Rate: 0.025 Mrad/hr
Total Dose: 0.550 Mrad
Temperature: 77°F
Atmosphere: Air

RESULTS:

Sample	Tensile Strength, psi	Elongation, %	Thickness, mil	Resistance, milliohm-inch^2	Resistance, milliohm-inch^2/mil
54-1, Top	1540	>100	1.5	15	10
	1390	>100	1.5	18	12
54-34, Bottom	1200	76	1.6	14	9
	1130	70	1.7	15	9
55-1, Middle	1295	75	1.4	15	11
	1395	>100	1.5	13	9
55-9, Top	1470	>100	1.5	28	19
	1640	>100	1.3	27	21
55-17, Bottom	1475	>100	1.3	26	20
	1475	>100	1.3	24	18
55-25, Top	1580	>100	1.5	40	25
	1740	>100	1.5	38	25
55-34, Middle	1085	84	1.5	21	14
	1085	45	1.5	18	12
Average	1393	-	1.5	22	15
Range	1085-1740	-	1.3-1.7	13-40	9-25

TABLE 12. SAMPLE NUMBERS 56 AND 57

GRAFTED AND CROSSLINKED

Experimental Conditions for Grafting:
Dose Rate: 0.021 Mrad/hr
Total Dose: 0.671 Mrad
Temperature: 125°F
Atmosphere: Nitrogen

Sample unavailable for crosslinking

RESULTS:

Sample	Tensile Strength, psi	Elongation, %	Thickness, mil	Resistance, milliohm-inch^2	Resistance, milliohm-inch^2/mil

Specimens were solid rods, no samples taken.

TABLE 13. SAMPLE NUMBERS 58 AND 59

GRAFTED AND CROSSLINKED

Experimental Conditions for Grafting:
Dose Rate: 0.021 Mrad/hr
Total Dose: 1.430 Mrad
Temperature: 125°F
Atmosphere: Air

Experimental Conditions for Crosslinking:
Dose Rate: 0.025 Mrad/hr
Total Dose: 0.550 Mrad
Temperature: 80°F
Atmosphere: Air

RESULTS:

Sample	Tensile Strength, psi	Elongation, %	Thickness, mil	Resistance milliohm-inch^2	Resistance milliohm-inch^2/mil
58-1,	1050	88	1.5	8	5
Middle	1250	>100	1.5	8	5
58-9,	1075	>100	2.0	10	5
Bottom	1310	>100	2.0	12	6
58-17,	810	>100	2.1	4	2
Top	835	>100	2.2	5	2
58-25,	1075	>100	2.3	9	4
Middle	1050	>100	2.2	8	4
58-34,	1150	>100	2.0	5	3
Middle	1070	>100	2.0	6	3
59-1,	1535	>100	1.4	11	8
Top	1200	>100	1.5	9	6
59-9,	1340	>100	1.8	12	7
Middle	1340	>100	1.8	8	4
59-17,	945	>100	1.8	4	2
Bottom	890	>100	1.8	4	2
59-25,	1020	>100	2.3	8	3
Top	1070	>100	2.4	7	3
59-34,	840	>100	2.0	3	2
Middle	680	>100	2.0	6	3
Average	1077	--	1.9	7	4
Range	680-1535		1.4-2.4	3-12	2-8

TABLE 14. SAMPLE NUMBERS 60 AND 61

GRAFTED ONLY

Experimental Conditions for Grafting:
Dose Rate: 0.021 Mrad/hr
Total Dose: 0.671 Mrad
Temperature: 125°F
Atmosphere: Air

RESULTS:

Sample	Tensile Strength, psi	Elongation, %	Thickness, mil	Resistance milliohm-inch^2	Resistance, milliohm-inch^2/mil
60-1,	1100	-	1.4	20	14
Top	940	70	1.5	19	13
60-9,	1415	>100	1.4	34	24
Middle	1240	90	1.6	34	21
60-17,	860	94	1.7	7	4
Bottom	755	85	1.8	10	6
60-25,	905	70	1.7	18	11
Top	845	70	1.8	19	11
60-34,	700	84	2.3	10	4
Middle	750	90	2.1	9	4
61-1,	920	-	1.2	15	12
Top	905	70	1.5	18	12
61-9,	710	65	1.7	22	13
Middle	775	70	1.7	21	12
61-17,	730	87	1.8	9	5
Bottom	555	70	1.7	8	5
61-25,	770	82	2.0	7	3
Middle	1050	84	2.0	7	3
61-35,	580	85	1.8	5	3
Bottom	780	>100	1.8	7	4
Average	865	-	1.7	15	9
Range	555-1415	-	1.2-2.3	5-34	3-24

TABLE 15. SAMPLE NUMBERS 62 AND 63

GRAFTED ONLY

Experimental Conditions for Grafting:
Dose Rate: 0.021 Mrad/hr
Total Dose: 1.430 Mrad
Temperature: 125°F
Atmosphere: Nitrogen

RESULTS:

Sample	Tensile Strength, psi	Elongation, %	Thickness, mil	Resistance, milliohm-inch^2	Resistance, milliohm-inch^2/mil
62(1)	1220	-	3.0	14	-
	400	85	3.0	13	-
	500	>100	3.5	11	-
	450	90	3.2	12	-
	530	>100	3.1	9	-
	600	70	2.8	12	-
	440	80	3.5	14	-
	-	-	-	14	-
	-	-	-	11	-
	-	-	-	9	-
62-34,	-	-	3.6	12	3
Top	-	-	3.6	11	3
63	NO SAMPLES TAKEN				

(1) Random sampling when possible.

TABLE 16. SAMPLE NUMBERS 64 AND 65

GRAFTED ONLY

Experimental Conditions for Grafting:
Dose Rate: 0.0125 Mrad/hr
Total Dose: 1.700 Mrad
Temperature: 125°F
Atmosphere: Air

RESULTS:

Sample	Tensile Strength, psi	Elongation, %	Thickness, mil	Resistance, milliohm-inch^2	Resistance, milliohm-inch^2/mil
64-1,	865	90	1.4	7	5
Top	765	80	1.4	6	4
64-9,	1045	>100	1.5	8	5
Middle	800	90	1.7	7	4
64-17,	880	70	1.5	15	10
Bottom	775	>100	1.7	15	9
64-25,	1065	>100	1.7	9	5
Middle	880	>100	1.5	8	5
64-34,	795	80	1.8	7	4
Bottom	855	90	1.8	9	5
65-1,	1100	95	1.5	11	7
Top	1050	>100	1.5	11	7
65-9,	745	>100	1.4	16	11
Middle	785	97	1.4	19	14
65-17,	865	>100	1.4	15	11
Bottom	865	>100	1.4	19	14
65-25,	880	90	1.5	11	7
Top	840	80	1.5	10	7
65-34,	740	50	1.7	6	4
Middle	630	70	1.5	5	3
Average	861	-	1.5	10	7
Range	630-1100	-	1.4-1.8	5-19	3-14

TABLE 17. SAMPLE NUMBERS 66 AND 67

GRAFTED ONLY

Experimental Conditions for Grafting:
Dose Rate: 0.0125 Mrad/hr
Total Dose: 0.800 Mrad
Temperature: 125°F
Atmosphere: Nitrogen

RESULTS:

Sample	Tensile Strength, psi	Elongation, %	Thickness, mil	Resistance, milliohm-inch^2	Resistance, milliohm-inch^2/mil
66-1,	680	90	1.7	4	3
Top	650	90	1.7	3	2
66-8,	470	>100	1.9	6	4
Top	300	65	2.0	5	3
67-1,	1100	>100	1.8	3	2
Top	795	>100	1.8	2	1

REMARKS: Specimens were very tacky, unable to obtain samples further in roll.

TABLE 18. SAMPLE NUMBERS 68 AND 69

GRAFTED AND CROSSLINKED

Experimental Conditions for Grafting:
Dose Rate: 0.0125 Mrad/hr
Total Dose: 1.700 Mrad
Temperature: 125°F
Atmosphere: Nitrogen

Experimental Conditions for Crosslinking:
Dose Rate: 0.025 Mrad/hr
Total Dose: 0.550 Mrad
Temperature: 80°F
Atmosphere: Air

RESULTS:

Sample	Tensile Strength, psi	Elongation, %	Thickness mil		Resistance, milliohm-inch^2		Resistance, milliohm-inch^2/mil	
			Before crosslinking	After crosslinking	Before crosslinking	After crosslinking	Before crosslinking	After crosslinking
68(1)	-	-	2.4	2.1	10	4	4	2
	-	-	2.5	2.3	8	4	3	2
	-	-	2.5	2.2	9	4	4	2
	-	-	2.4	2.2	7	4	3	2
	-	-	2.1	2.2	7	5	3	2
	-	-	2.0	2.2	6	5	3	2
	-	-	2.3	2.2	6	5	3	2
	-	-	2.6	2.2	7	4	3	2
	-	-	2.6	2.0	7	5	3	2
	-	-	2.5	2.0	6	5	3	2
69	DISCARDED, Unable to obtain samples.							
Average	-	-	2.4	2.2	7	5	3	2
Range	-	-	2.0-2.6	2.0-2.3	6-10	4-5	3-4	-

(1) Random sampling when possible.

TABLE 19. SAMPLE NUMBERS 70 AND 71

GRAFTED AND CROSSLINKED

Experimental Conditions for Grafting:
Dose Rate: 0.0125 Mrad/hr
Total Dose: 0.800 Mrad
Temperature: 125° F
Atmosphere: Air

Experimental Conditions for Crosslinking:
Dose Rate: 0.025 Mrad/hr
Total Dose: 0.550 Mrad
Temperature: 80°F
Atmosphere: Air

RESULTS:

Sample	Tensile Strength, psi	Elongation, %	Thickness, mil	Resistance, milliohm-inch^2	Resistance, milliohm-inch^2/mil
10-1,	1065	>100	1.2	12	10
Top	1100	>100	1.3	11	8
70-9,	1000	>100	2.0	13	7
Middle	925	>100	1.9	15	8
70-17,	910	>100	2.0	15	8
Bottom	715	80	2.0	16	8
70-25,	1045	>100	2.0	13	7
Middle	1040	>100	1.9	10	5
70-34,	980	>100	1.8	6	3
Bottom	980	95	1.8	8	4
71-1,	785	65	1.4	12	9
Top	1180	84	1.4	14	10
71-9,	990	90	2.0	22	10
Middle	990	84	2.0	17	9
71-17,	1070	>100	2.0	12	6
Bottom	935	90	2.0	13	8
71-25,	915	89	1.8	9	5
Top	980	90	1.8	9	5
71-34,	2260	>100	1.3	600	460
Middle	1235	>100	1.5	21	14
* Average	987	-	1.8	13	8
* Range	715 1235	-	1.2-2.0	6-22	3-14

* Does not include high resistance specimen.

TABLE 20. SUMMARY OF RESISTANCE VALUES FROM FACTORIAL EXPERIMENT*

		Grafting Dose Rate: 0.021 Mrad				Grafting Dose Rate: 0.0125 Mrad			
		Total Dose: 1.430 Mrad		Total Dose: 0.671 Mrad		Total Dose: 1.700 Mrad		Total Dose: 0.800 Mrad	
		O_2	N_2	O_2	N_2	O_2	N_2	O_2	N_2
Grafted Only	77°F	12-58 (27)			9-21 (13)		16-34 (24)	10-133 (34)	
	125°F		9-14 (12)	5-34 (15)		5-19 (10)			2-6 (4)
Grafted then Crosslinked	77°F		9-38 (22)	10-37 (20)		20-71 (43)			13-40 (22)
	125°F	3-12 (7)			no samples		4-5 (5)	6-22 (13)	

*Values are in milliohm-inch^2. Values in parenthesis are average values, and the other values are the ranges obtained. When high resistance values occurred in the outermost few feet of the roll, these values were disregarded.

TABLE 21. GRAFTING CONDITIONS AND EXOTHERM

Sample No.	Atmosphere	Dose Rate, Mrad/hr	Total Dose, Mrad	Time to Exotherm	Duration of Exotherm, (Maximum Exotherm, °F)
T_1 = 77°F					
36	O_2	0.021	1.430	No exotherm	
37	O_2	0.021	1.430	No exotherm	
38	N_2	0.021	1.430	8.0 hr	8 hr (18)
39	N_2	0.021	1.430	8.0 hr	8 hr (18)
44	N_2	0.021	0.671	No exotherm	
45	N_2	0.021	0.671	No exotherm	
46	O_2	0.021	0.671	No exotherm	
47	O_2	0.021	0.671	No exotherm	
48	N_2	0.0125	1.700	No exotherm	
49	N_2	0.0125	1.700	No exotherm	
50	O_2	0.0125	1.700	No exotherm	
51	O_2	0.0125	1.700	No exotherm	
52	O_2	0.0125	0.800	No exotherm	
53	O_2	0.0125	0.800	No exotherm	
54	N_2	0.0125	0.800	No exotherm	
55	N_2	0.0125	0.800	No exotherm	
T_2 = 125°F					
56	N_2	0.021	0.671	0.3 hr	5 hr (75)
57	N_2	0.021	0.671	0.6 hr	6 hr (65)
58	O_2	0.021	1.430	10.0 hr	9 hr (51)
59	O_2	0.021	1.430	10.0 hr	9 hr (21)
60	O_2	0.021	0.671	10.0 hr	9 hr (29)
61	O_2	0.021	0.671	10.0 hr	9 hr (11)
62	N_2	0.021	1.430	1.0 hr	8 hr (53)
63	N_2	0.021	1.430	0.6 hr	9 hr (55)
64	O_2	0.0125	1.700	8.0 hr	2 hr (10)
65	O_2	0.0125	1.700	8.0 hr	2 hr (10)
66	N_2	0.0125	0.800	0.6 hr	4 hr (10)
67	N_2	0.0125	0.800	0.6 hr	5 hr (55)
68	N_2	0.0125	1.700	0.6 hr	5 hr (50)
69	N_2	0.0125	1.700	0.6 hr	4 hr (60)
70	O_2	0.0125	0.800	No exotherm	
71	O_2	0.0125	0.800	No exotherm	

TABLE 22. EFFECT OF STERILIZATION ON RESISTANCE OF TEST PLAN SAMPLES

Sample	Thickness, mil		Resistance, milliohm - inch^2		Resistance, milliohm - inch^2/mil	
	Before	After	Before	After	Before	After
37 1	1.3	1.6	16	9	12	6
38 32	1.5	1 6	27	11	18	7
44 10	1.6	2.0	12	15	8	8
46 34	1.7	1.8	15	5	9	3
48-1	1.5	2.0	16	7	11	4
50 34	1.5	1.6	36	16	24	10
52-13	1.2	2.0	109	9	91	5
54 34	1.6	1.6	14	9	9	6
58-17	2.1	2.2	5	6	2	3
60 17	1.8	2.0	10	8	6	4
62 ?*	3.0	3.0	12	4	4	1
64 17	1.5	1.7	15	10	10	6
66-1	1.7	1.7	4	13	2	8
68 - ?*	2.2	2.2	5	4	2	2
70-25	1.9	1.7	10	5	5	3

* Random sample from the roll

REMARKS: Samples were sterilized for 64 hours at 135°C in 40 wt % KOH.

TABLE 23. SAMPLE NUMBERS 81 THROUGH 88

GRAFTED AND CROSSLINKED

Grafting Solution Composition:	Experimental Conditions for Grafting:	
25 wt % Acrylic acid	Dose Rate:	0.021 Mrad/hr
70 wt % Benzene	Total Dose:	1.430 Mrad
5 wt % Carbon tetrachloride	Temperature:	89°F
	Atmosphere:	Nitrogen

RESULTS:

Sample	Resistance, milliohm-inch^2
81-1	9
	9
81-34	12
	12
82-1	9
	9
82-34	30
	18
83-1	12
	7
83-34	9
	10
84-1	12
	7
84-34	9
	8
85-1	13
	12
85-34	8
	9
86-1	11
	10
86-34	10
	10
87-1	14
	11
87-34	5
	13
88-1	17
	16
88-34	14
	17
Average	12
Range	5-30

Exotherm Data for Grafting:	Samples:	82	86	88
Time to exotherm, hr:		2	4	4
Duration of exotherm, hr:		18	16	16
Maximum exotherm, °F:		23	31	11

TABLE 24. SAMPLE NUMBERS 105 THROUGH 112

GRAFTED AND CROSSLINKED

Grafting Solution Composition:	Experimental Conditions for Grafting:	
25 wt % Acrylic acid	Dose Rate:	0.0210 Mrad/hr
70 wt % Benzene	Total Dose:	1.430 Mrad
5 wt % Carbon tetrachloride	Temperature:	86 °F
	Atmosphere:	Nitrogen

RESULTS:

Sample	Resistance, milliohm-inch^2
105-1	8
	8
105-34	7
	11
106-1	8
	7
106-34	6
	8
107 - No samples taken	
108-1	10
	11
108-34	8
	8
109-1	10
	10
109-34	14
	14
110-1	10
	12
110-34	11
	9
111-1	7
	7
111-34	15
	11
112-1	9
	8
112-34	11
	8
Average	10
Range	6-15

Exotherm Data for Grafting:	Samples: 105	108	112
Time to exotherm, hr:	2	2	2
Duration of exotherm, hr:	20	18	19
Maximum exotherm, °F:	17	14	12

TABLE 25. SAMPLE NUMBER 73

GRAFTED AND CROSSLINKED

Grafting Solution Composition:

25 wt % Acrylic acid
70 wt % Benzene
5 wt % Carbon tetrachloride

Experimental Conditions for Grafting:

Dose Rate: 0.021 Mrad/hr
Total Dose: 1.430 Mrad
Temperature: 125°F
Atmosphere: Air

RESULTS:

Sample	Thickness, mil	Resistance, milliohm-inch^2	Resistance, milliohm-inch^2/mil
73-1, Top	1.3	18	14
	1.5	18	12
73-9, Middle	1.6	7	4
	1.7	6	4
73-17, Bottom	1.7	7	4
	1.8	7	4
73-25, Top	1.9	6	3
	1.8	5	3
73-34, Middle	1.9	4	2
	1.9	5	3
Average	1.7	8	5
Range	1.3 - 1.9	4 - 18	2 - 14

Exotherm Data for Grafting:

Time to exotherm, hr: 5
Duration of exotherm, hr: 19
Maximum exotherm, °F: 15

TABLE 26. SAMPLE NUMBER 74

GRAFTED AND CROSSLINKED

Grafting Solution Composition:

25 wt % Acrylic acid
70 wt % Benzene
5 wt % Carbon tetrachloride

Experimental Conditions for Grafting:

Dose Rate: 0.021 Mrad/hr
Total Dose: 1.430 Mrad
Temperature: 125°F
Atmosphere: Air

RESULTS:

Sample	Thickness, mil	Resistance, milliohm-inch^2	Resistance, milliohm-inch^2/mil
74-1, Middle	1.5	11	7
	1.5	12	8
74-9, Bottom	1.5	16	11
	1.5	17	11
74-17, Top	1.9	12	6
	2.0	8	4
74-25, Middle	1.9	8	4
	1.9	8	4
74-34, Middle	2.2	6	3
	2.2	5	2
Average	1.8	10	6
Range	1.5 - 2.2	5 - 17	2 - 11

Exotherm Data for Grafting:

Time to exotherm, hr: 5
Duration of exotherm, hr: 19
Maximum exotherm, °F: 21

TABLE 27. SAMPLE NUMBER 15

CROSSLINKED AND GRAFTED

Grafting Solution Composition:

25 wt % Acrylic acid
70 wt % Benzene
5 wt % Carbon tetrachloride

Experimental Conditions for Grafting:

Dose Rate: 0.021 Mrad/hr
Total Dose: 1.430 Mrad
Temperature: 90°F
Atmosphere: Nitrogen

RESULTS:

Sample	Tensile Strength psi	Elongation, %	Thickness, mil	Resistance, milliohm-inch^2	Resistance, milliohm-inch^2/mil
15-1,	1690	70	1.2	1175	980
Top	1860	92	1.1	>3000	-
15-11,	2060	>100	-	>3000	-
Middle	1915	85	-	>3000	-
15-23,	2130	>100	-	>3000	-
Bottom	1955	>100	-	>3000	-
15-34,	1600	85	1.5	150	100
Top	1745	90	1.7	10	6
Average	1870	-	-	-	-
Range	1600-2130				

Exotherm Data for Grafting:

Time to exotherm, hr: 3
Duration of exotherm, hr: 17
Maximum exotherm, °F: 21

TABLE 28. SAMPLE NUMBER 12

CROSSLINKED AND GRAFTED

Grafting Solution Composition:

25 wt % Acrylic acid
70 wt % Benzene
5 wt % Carbon tetrachloride

Experimental Conditions for Grafting:

Dose Rate: 0.021 Mrad/hr
Total Dose: 0.671 Mrad
Temperature: 125°F
Atmosphere: Air

RESULTS:

Sample	Tensile Strength, psi	Elongation, %	Thickness, mil	Resistance, milliohm-inch^2	Resistance, milliohm-inch^2/mil
12-1,	3100	>100	1.0	>3000	-
Bottom	3480	>100	1.0	>3000	-
12-9,	3360	>100	1.0	>3000	-
Middle	3240	>100	1.0	>3000	-
12-17,	2670	>100	1.0	>3000	-
Top	2640	>100	1.1	>3000	-
12-25,	1960	>100	1.5	>3000	-
Middle	1860	>100	1.6	>3000	-
12-34,	2050	>100	1.0	400	400
Bottom	2080	>100	1.0	77	77

Exotherm Data for Grafting:

Time to exotherm, hr: 7
Duration of exotherm, hr: 5
Maximum exotherm, °F: 5

TABLE 29. SAMPLE NUMBER 13

CROSSLINKED AND GRAFTED

Grafting Solution Composition:

25 wt % Acrylic acid
70 wt % Benzene
5 wt % Carbon tetrachloride

Experimental Conditions for Grafting:

Dose Rate: 0.021 Mrad/hr
Total Dose: 0.671 Mrad
Temperature: 125°F
Atmosphere: Nitrogen

RESULTS:

Sample	Tensile Strength, psi	Elongation, %	Thickness, mil	Resistance, milliohm-inch^2	Resistance, milliohm-inch^2/mil
13-1,	1510	>100	1.3	>3000	-
Top	1380	83	1.2	>3000	-
13-9,	890	88	2.2	>3000	-
Middle	970	85	2.2	>3000	-
13-17,	775	>100	1.7	6	4
Bottom	855	>100	1.8	8	4
13-25,	945	>100	2.1	8	4
Top	880	>100	2.0	13	7
13-34,	1040	>100	2.0	9	5
Middle	1070	>100	2.0	4	2

Exotherm Data for Grafting:

Time to exotherm, hr: 2
Duration of exotherm, hr: 10
Maximum exotherm, °F: 16

TABLE 30. SAMPLE NUMBER 16

CROSSLINKED AND GRAFTED
(5% KOH wash before grafting)

Grafting Solution Composition:

25 wt % Acrylic acid
70 wt % Benzene
5 wt % Carbon tetrachloride

Experimental Conditions for Grafting:

Dose Rate: 0.021 Mrad/hr
Total Dose: 1.430 Mrad
Temperature: 90°F
Atmosphere: Nitrogen

RESULTS:

Sample	Tensile Strength, psi	Elongation, %	Thickness, mil	Resistance, milliohm-inch^2	Resistance, milliohm-inch^2/mil
16-1,	1125	95	1.7	8	5
Top	885	50	1.8	10	6
16-11,	1375	>100	1.6	825	515
Middle	1530	>100	1.7	391	230
16-23,	1550	90	1.4	860	
Bottom	1500	>100	1.5	165	615
					111
16-34,	1715	>100	1.2	169	140
Top	1695	>100	1.3	117	90
Average	1430	-	1.5	318	214
Range	885-1715	-	1.2 - 1.8	8 - 860	5 - 615

Exotherm Data for Grafting:

Time to exotherm, hr: 3
Duration of exotherm, hr: 17
Maximum exotherm, °F: 30

TABLE 31. SAMPLE NUMBER 91

GRAFTED ONLY

Grafting Solution Composition:

5 wt % Acrylic acid
94 wt % Benzene
1 wt % Carbon tetrachloride

Experimental Conditions for Grafting:

Dose Rate: 0.021 Mrad/hr
Total Dose: 1.430 Mrad
Temperature: 90°F
Atmosphere: Nitrogen

RESULTS:

Sample	Tensile Strength, psi	Elongation, %	Thickness, mil	Resistance, milliohm-inch^2	Resistance, milliohm-inch^2/mil
91-1,	1350	65	1.2	193	138
Top	1320	98	1.4	>3000	-
91-11,	1310	>100	1.9	>3000	-
Middle	1785	>100	1.9	>3000	-
91-23,	1590	>100	1.7	>3000	-
Bottom	1250	>100	1.7	>3000	-
91-34,	2500	>100	1.1	>3000	-
Top	2340	>100	1.1	>3000	-
Average	1680	-	1.5	-	-
Range	1250-2500	-	1.1 - 1.9	-	-

Exotherm Data for Grafting:

Time to exotherm, hr: 3
Duration of exotherm, hr: 17
Maximum exotherm, °F: 8

TABLE 32. SAMPLE NUMBER 92

GRAFTED ONLY

Grafting Solution Composition:

10 wt % Acrylic acid
88 wt % Benzene
2 wt % Carbon tetrachloride

Experimental Conditions for Grafting:

Dose Rate: 0.021 Mrad/hr
Total Dose: 1.430 Mrad
Temperature: 90°F
Atmosphere: Nitrogen

RESULTS:

Sample	Tensile Strength psi	Elongation %	Thickness, mil	Resistance, milliohm-inch^2	Resistance, milliohm-inch^2/mil
92-1,	945	>100	1.1	32	28
Top	1000	>100	1.3	15	12
92-11,	1335	>100	1.3	15	12
Middle	1415	>100	1.3	28	21
92-23,	1000	>100	2.3	>3000	-
Bottom	930	>100	2.5	780	311
92-34,	1470	>100	1.3	28	22
Top	1310	92	1.4	75	54
Average*	1175	-	1.6	139	66
Range*	930-1470	-	1.1 - 2.5	15 - 780	12 - 311

Exotherm Data for Grafting:

Time to exotherm, hr: 3
Duration of exotherm, hr: 17
Maximum exotherm, °F: 8

* Does not include high resistance values.

TABLE 33. SAMPLE NUMBER 93

GRAFTED ONLY

Grafting Solution Composition:

15 wt % Acrylic acid
82 wt % Benzene
3 wt % Carbon tetrachloride

Experimental Conditions for Grafting:

Dose Rate: 0.021 Mrad/hr
Total Dose: 1.430 Mrad
Temperature: 90°F
Atmosphere: Nitrogen

RESULTS:

Sample	Tensile Strength, psi	Elongation, %	Thickness, mil	Resistance, milliohm-inch^2	Resistance, milliohm-inch^2/mil
93-1,	1100	93	1.4	21	15
Top	1200	>100	1.4	18	13
93-11,	1430	>100	1.4	28	20
Middle	1235	85	1.4	66	47
93-23,	1230	>100	1.7	66	39
Bottom	1200	97	1.7	55	32
93-34,	1275	>100	1.4	23	16
Top	1245	>100	1.5	22	15
Average	1240	-	1.5	37	25
Range:	1100-1430	-	1.4 - 1.7	18 - 66	13 - 47

Exotherm Data for Grafting:

Time to exotherm, hr: 1
Duration of exotherm, hr: 19
Maximum exotherm, °F: 14

TABLE 34. SAMPLE NUMBER 94

GRAFTED ONLY

Grafting Solution Composition:

20 wt % Acrylic acid
76 wt % Benzene
4 wt % Carbon tetrachloride

Experimental Conditions for Grafting:

Dose Rate: 0.021 Mrad/hr
Total Dose: 1.430 Mrad
Temperature: 90°F
Atmosphere: Nitrogen

RESULTS:

Sample	Tensile Strength, psi	Elongation, %	Thickness, mil	Resistance, milliohm-inch^2	Resistance, milliohm-inch^2/mil
94-1,	1065	95	1.4	9	6
Top	1030	95	1.3	9	7
94-11,	1200	>100	1.4	30	21
Middle	1135	95	1.5	20	13
94-23,	1165	>100	1.7	25	15
Middle	1165	>100	1.6	20	12
94-34,	1500	>100	1.3	16	12
Top	1135	>100	1.5	14	9
Average	1175	-	1.5	18	12
Range	1030-1500	-	1.3 - 1.7	9 - 30	6 - 21

Exotherm Data for Grafting:

Time to exotherm, hr: 1
Duration of exotherm, hr: 19
Maximum exotherm, °F: 25

TABLE 35. SAMPLE NUMBERS 101 AND 102

GRAFTED ONLY

Grafting Solution Composition:

25 wt % Acrylic acid
75 wt % Benzene

Experimental Conditions for Grafting:

Dose Rate:	0.021 Mrad/hr
Total Dose:	1.430 Mrad
Temperature:	125°F
Atmosphere:	Air

RESULTS:

Sample	Thickness, mil	Resistance, milliohm-inch^2	Resistance, milliohm-inch^2/mil
101-1, Top	0.9	>3000	-
	1.0	>3000	-
	1.0	>3000	-
101-7, Middle	2.0	5	3
	2.0	2	1
	1.8	4	2
101-23, Bottom	2.4	4	2
	2.2	4	2
	2.4	4	2
101-33, Middle	2.2	7	3
	2.1	5	2
	2.2	2	1
102-?	1.2	8	7
	1.1	5	5
	1.3	9	7
	1.5	6	4
	1.0	>3000	-
	1.4	8	6
	1.5	6	4
	2.5	4	2
	2.5	5	2

Exotherm Data for Grafting:	Samples: 101	102
Time to exotherm, hr:	4	3
Duration of exotherm, hr:	8	9
Maximum exotherm, °F:	13	31

REMARKS: Film came apart in 5% KOH
Tensile strength for 101-23, Bottom: 270 & 500 psi, 70 & 85% Elongation
Tensile strength for 101-33, Middle: 660 psi & 70% Elongation.

TABLE 36. SAMPLE NUMBER 78

GRAFTED ONLY

Grafting Solution Composition:

25 wt % Acrylic acid
72.5 wt % Benzene
2.5 wt % Carbon tetrachloride

Experimental Conditions for Grafting:

Dose Rate: 0.021 Mrad/hr
Total Dose: 1.430 Mrad
Temperature: 125°F
Atmosphere: Air

RESULTS:

Sample	Tensile Strength psi	Elongation, %	Thickness, mil	Resistance, milliohm-inch^2	Resistance, milliohm-inch^2/mil
78-1,	740	>100	1.7	4	2
Bottom	710	>100	1.6	5	3
78-9,	790	>100	2.1	4	2
Middle	325	55	2.1	5	2
78-17,	425	80	2.4	3	1
Top	370	75	2.3	3	1
78-25,	640	>100	2.5	2	1
Top	760	>100	2.5	3	1
78-34,	525	>100	1.8	6	3
Middle	750	>100	1.6	4	2
Average	605	-	2.1	4	2
Range	325-790	-	1.6 - 2.5	2 - 6	1 - 3

Exotherm Data for Grafting:

Time to exotherm, hr: 5
Duration of exotherm, hr: 19
Maximum exotherm, °F: 31

TABLE 37. SAMPLE NUMBERS 97 AND 98

GRAFTED ONLY

Grafting Solution Composition:	Experimental Conditions for Grafting:	
25 wt % Acrylic acid	Dose Rate:	0.021 Mrad/hr
72.5 wt % Benzene	Total Dose:	1.430 Mrad
2.5 wt % Carbon tetrachloride	Temperature:	125°F
	Atmosphere:	Air

RESULTS:

Sample	Thickness, mil	Resistance, milliohm-inch^2	Resistance, milliohm-inch^2/mil

NO SAMPLES TAKEN

Exotherm Data for Grafting:	Samples:	97	98
Time to exotherm, hr:		4	4
Duration of exotherm, hr:		4	4
Maximum exotherm, °F:		43	43

REMARKS: Film had no strength, unable to separate from paper.

TABLE 38. SAMPLE NUMBERS 99 AND 100

GRAFTED ONLY

Grafting Solution Composition:

25 wt % Acrylic acid
72.5 wt % Benzene
2.5 wt % Carbon tetrachloride

Experimental Conditions for Grafting:

Dose Rate: 0.021 Mrad/hr
Total Dose: 1.430 Mrad
Temperature: 125°F
Atmosphere: Air

RESULTS:

Sample	Thickness, mil	Resistance, milliohm-inch^2	Resistance, milliohm-inch^2/mil
99-4, Middle	1.6	11	7
	1.6	16	10
	1.8	17	9
99-11, Bottom	2.0	11	5
	2.0	11	5
	2.0	12	6
99-23, Middle	2.2	10	5
	2.4	8	3
	2.2	10	5
99-33, Top	1.3	>3000	-
	2.2	4	2
	1.2	>3000	-
100-1, Middle	1.4	21	15
	1.4	20	14
	1.4	22	16
100-11, Bottom	1.7	14	8
	1.6	9	6
	1.8	11	6
100-23, Middle	1.6	8	5
	1.5	6	4
	1.4	9	6
100-33, Top	1.9	10	5
	2.0	16	8
	1.9	14	7

Exotherm Data for Grafting:	Samples: 99	100
Time to exotherm, hr:	4	4
Duration of exotherm, hr:	8	8
Maximum exotherm, °F:	13	13

TABLE 39. SAMPLE NUMBER 79

GRAFTED ONLY

Grafting Solution Composition:

25 wt % Acrylic acid
65 wt % Benzene
10 wt % Carbon tetrachloride

Experimental Conditions for Grafting:

Dose Rate: 0.021 Mrad/hr
Total Dose: 1.430 Mrad
Temperature: 125 °F
Atmosphere: Air

RESULTS:

NO SAMPLES TAKEN

Exotherm Data for Grafting:

Time to exotherm, hr: 5
Duration of exotherm, hr: 19
Maximum exotherm, °F: 49

REMARKS: Film had no strength, unable to separate from paper.

TABLE 40. SAMPLE NUMBER 103

GRAFTED ONLY

Grafting Solution Composition:

25 wt % Acrylic acid
37.5 wt % Benzene
37.5 wt % Xylene

Experimental Conditions for Grafting:

Dose Rate: 0.021 Mrad/hr
Total Dose: 1.430 Mrad
Temperature: 125°F
Atmosphere: Air

RESULTS:

Sample	Thickness, mil	Resistance, milliohm-inch^2	Resistance, milliohm-inch^2/mil
103-1, Bottom	1.0	3000	-
	1.1	3000	-
	1.0	3000	-
103-6, Middle	1.8	14	8
	1.8	13	7
	1.7	15	9
103-16, Top	1.9	6	3
	1.8	8	4
	1.9	11	6
103-24, Middle	1.9	7	4
	1.9	9	5
	1.9	8	4
103-32, Bottom	1.9	3	2
	1.8	5	3
	1.9	6	3

Exotherm Data for Grafting:

Time to exotherm, hr: 3
Duration of exotherm, hr: 9
Maximum exotherm, °F: 6

TABLE 41. SAMPLE NUMBER 104

GRAFTED ONLY

Grafting Solution Composition:

25 wt % Acrylic acid
60 wt % Benzene
15 wt % Xylene

Experimental Conditions for Grafting:

Dose Rate:	0.021 Mrad/hr
Total Dose:	1.430 Mrad
Temperature:	125°F
Atmosphere:	Air

RESULTS:

Sample	Thickness, mil	Resistance, milliohm-inch^2	Resistance, milliohm-inch^2/mil
104-1, Top	1.0	>3000	-
	1.0	>3000	-
	1.0	>3000	-
104-8, Middle	2.0	7	4
	2.0	8	4
	1.9	7	4
104-16, Bottom	1.9	7	4
	1.9	8	4
	1.9	9	5
104-24, Top	2.1	6	3
	2.0	4	2
	2.0	6	3
104-32, Middle	1.0	>3000	-
	1.0	>3000	-
	1.0	>3000	-

Exotherm Data for Grafting:

Time to exotherm, hr:	4
Duration of exotherm, hr:	8
Maximum exotherm, °F:	13

TABLE 42. SAMPLE NUMBER 76

GRAFTED ONLY

Grafting Solution Composition:

25 wt % Acrylic acid
70 wt % Xylene
5 wt % Carbon tetrachloride

Experimental Conditions for Grafting:

Dose Rate: 0.021 Mrad/hr
Total Dose: 1.430 Mrad
Temperature: 125°F
Atmosphere: Air

RESULTS:

Sample	Tensile Strength, psi	Elongation, %	Thickness, mil	Resistance, milliohm-inch^2	Resistance, milliohm-inch^2/mil
76-1,	735	>100	1.8	16	9
Top	690	80	1.6	17	11
76-9,	1195	>100	1.8	21	12
Middle	1160	>100	1.8	24	13
76-17,	560	80	1.9	1	<1
Bottom	605	80	1.8	<1	<1
76-25,	720	50	1.9	4	2
Top	605	75	1.9	4	2
76-34,	575	80	2.1	5	2
Middle	665	92	2.1	5	2
Average	750	-	1.9	10	5
Range	560-1195	-	1.6 - 2.1	<1 - 24	<1 - 13

Exotherm Data for Grafting:

Time to exotherm, hr: 5
Duration of exotherm, hr: 19
Maximum exotherm, °F: 31

TABLE 43. SAMPLE NUMBER 77

GRAFTED ONLY

Grafting Solution Composition:

25 wt % Acrylic acid
70 wt % Xylene
5 wt % Carbon tetrachloride

Experimental Conditions for Grafting:

Dose Rate: 0.021 Mrad/hr
Total Dose: 1.430 Mrad
Temperature: 125°F
Atmosphere: Nitrogen

RESULTS:

Sample	Tensile Strength, psi	Elongation, %	Thickness, mil	Resistance, milliohm-inch^2	Resistance, milliohm-inch^2/mil
77-7,	510	90	2.4	16	7
Bottom	645	>100	2.6	17	7
77-15,	630	>100	2.8	26	9
Middle	605	72	2.5	37	15
77-23,	545	94	2.5	5	2
Top	545	90	2.3	<1	<1
77-31,	840	>100	2.1	13	6
Middle	705	98	2.2	15	7
Average	630	-	2.4	16	7
Range	510-840	-	2.1 - 2.8	<1 - 37	<1 - 15

Exotherm Data for Grafting:

Time to exotherm, hr: 2.5
Duration of exotherm, hr: 22
Maximum exotherm, °F: 45

APPENDIX A

Irradiation Techniques

CROSSLINKING PROCEDURE

1. A thirty-foot length of film is backed with absorbent crepe paper toweling and rolled onto a one-quarter inch aluminum pipe which is capped at one end.

2. The roll of film is placed in a hydrometer jar (75 mm x 550 mm) and covered with a one-percent (by volume) solution of divinylbenzene in a mixture of one part of benzene in 98 parts of methanol by volume.

3. The jar is alternately evacuated and allowed to return to atmospheric pressure until air bubbles can no longer be removed from the roll of film by evacuation.

4. The top of the jar is covered with aluminum foil to retard evaporation of the solution, and the film is allowed to equilibrate for at least 24 hours.

5. The jar and roll of film are exposed to a uniform cobalt-60 source adjusted to give a dose rate of 0.025 Mrad per hour until a total dose of 0.55 Mrad is obtained.

6. The jar is removed from the source and allowed to stand at least 24 hours. The film is unrolled and washed by passing it through benzene at a rate that permits a one-minute contact time with the benzene. The washed film is backed with paper toweling, rolled and permitted to dry.

GRAFTING PROCEDURE

1. A thirty-foot length of film is backed with absorbent crepe paper toweling and rolled onto a one-quarter inch aluminum pipe which is capped at one end.

2. The roll of film is placed in a hydrometer jar (75 mm x 550 mm) and covered with an acrylic acid solution having a composition of 25% acrylic acid, 70% benzene and 5% carbon tetrachloride by weight.

3. The jar is alternately evacuated and allowed to return to atmospheric pressure until air can no longer be removed from the roll of film by evacuation.

4. The top of the jar is covered with aluminum foil to retard evaporation of the solution, and the film is allowed to equilibrate for at least 24 hours.

5. The jar and roll of film are exposed to a uniform cobalt-60 source adjusted to give a dose rate of 0.021 Mrad per hour until a total dose of 1.430 Mrad is obtained.

6. The jar is removed from the source and allowed to stand at least 24 hours. The film is unrolled and washed in hot (80°C) aqueous 5% potassium hydroxide solution for one hour.

7. The film is then washed in hot (80°C) water for one hour and allowed to dry on paper toweling.